National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1751

Gallium Melting-Point Standard

Certified Melting-Point Temperature 29.764 6 °C ± 0.000 07 °C

International Temperature Scale of 1990 (ITS-90)

This Standard Reference Material (SRM) is intended primarily for use as one of the defining fixed points of the International Temperature Scale of 1990 (ITS-90) [1,2]. The melting point is realized as the plateau temperature of the melting curve of slowly-melting, high-purity (mass fraction ≥ 99.9999 % pure) gallium. Each unit of SRM 1751 consists of 200 g of gallium sealed in an argon atmosphere in a plastic bottle.

Certified Value and Confidence Limit: Randomly selected SRM 1751 units (10 % of available units) were used to fabricate three gallium melting-point cells (Ga 981, Ga 982, Ga 983) that were used in conjunction with a standard platinum resistance thermometer (SPRT) to experimentally evaluate melting-curve plateaus and the melting-point temperature of SRM 1751 with respect to the NIST laboratory-reference gallium melting-point cell. The expanded uncertainty ($k = 2$) of 0.000 07 °C is assigned to the melting-point temperature.

Certified Melting-Point Temperature: 29.764 6 °C ± 0.000 07 °C.

As shown in Figure 1, the temperature range of melting of the samples tested of this current lot of material is expected to be less than 0.000 01 °C. As shown in Figure 2, the average melting-curve plateau temperature is expected to differ among different samples by not more than ± 0.000 01 °C. A mathematical evaluation of the manufacturer sample assay was used to estimate the effect of impurities on the melting-point realization temperature [2].

Expiration of Certification: The certification of **SRM 1751 (Lot No. 9800763)** is valid indefinitely within the measurement uncertainties specified, provided the SRM is used in accordance with the instructions given in the "Notice and Warnings to Users" section of this certificate. The certification is nullified if the SRM or SRM container is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Temperature measurements of the gallium melting-point standard were performed by G.F. Strouse of the NIST Process Measurements Division.

The support aspects involved with issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division

James R. Whetstone, Chief
Process Measurements Division

Gaithersburg, MD 20899
Certificate Issue Date: 17 March 2004

John Rumble, Jr, Chief
Measurement Services Division

Source of Material: The 30 kg of gallium metal **(Lot No. 9800763)** for this SRM was obtained from Rhodia-Chimie[1] of France.

The emission spectrographic assay of the gallium metal for this SRM shows the total impurity level to be 0.044 : g/g, resulting from 0.001 : g/g of aluminum (Al), 0.002 : g/g of chlorine (Cl), 0.005 : g/g of copper (Cu), 0.004 : g/g of iron (Fe), 0.01 : g/g of nickel (Ni), 0.01 : g/g of lead (Pb), 0.001 : g/g of titanium (Ti), 0.01 : g/g of thallium (Tl) and 0.001 : g/g of vanadium (V).

NOTICE AND WARNING TO USERS

Storage: SRM 1751 is package in a sealed argon atmosphere. An unopened unit may be stored in a normal laboratory environment. An opened unit should be stored in a dry box containing an inert gas (preferably argon) to prevent oxidation and possible contamination.

Handling: Physical handling of high-purity material is apt to introduce contamination. This SRM is provided in a container to minimize the need for handling during the construction of a gallium melting-point cell. However, every possible effort should be made to maintain purity of this SRM through the use of disposable powder-free plastic gloves while handling. It is suggested that an argon-filled glove box be used when transferring the gallium from the original container to the user's receptacle. It is important to note that liquid gallium is corrosive to other metals and proper safety cautions should be observed.

INSTRUCTIONS FOR USE

In assigning a temperature value to the realization of the gallium melting point for calibration purposes, a correction of -1.2×10^{-3} °C/m must be applied for the average depth of immersion of the thermometer sensing element below the surface of the metal. Also, if the pressure over the cell during the measurements is not controlled to 101 325 Pa, a correction of -2.0×10^{-8} °C/Pa must be made for the difference in pressure.

A detailed description on the fabrication of a gallium thermometric fixed-point cell is found in references 2 and 3. Additionally, a suggested uncertainty budget for the use of a cell containing this SRM is given in reference 2. Values left blank in the suggested uncertainty budget must be determined by the end user in their laboratory using their own equipment and personnel.

PREPARATION AND CERTIFICATION MEASUREMENTS

Method of Preparation: The thermal tests for the certification of this SRM were performed on three melting-point cells (Ga 981, Ga 982, Ga 983) prepared in a manner described in reference 1. Each cell contains approximately 1000 g of gallium from randomly selected units of **Lot No. 9800763**.

Measurement Techniques: The experimental measurements used to evaluate SRM 1751 included a commercially-available 9.5 digit ac resistance ratio bridge operating at a frequency of 30 Hz, a thermostatically-controlled (25 ± 0.01) °C ac/dc reference resistor and a 25.5 Ω SPRT. Melting plateau measurements were made with an excitation current of 1 mA. Direct comparison measurements were made at 1 mA and 1.414 mA for extrapolation of the measured resistance ratio to 0 mA. Data acquisition was controlled over an IEEE-488 bus.

The furnaces used to realize the gallium melting-point consist of a closed-bottom cylindrical-tube aluminum core with a non-inductively, single-layer, bifilarly wound heater on the outside of the core. The furnaces are designed to provide about a 1 mm annular space between the Ga cell and the inner wall of the aluminum core. The aluminum core is filled with mineral oil to improve thermal contact between the cell and the furnace core. An immersion heater operating at a temperature of 40 °C is used to establish the inner liquid-solid interface.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

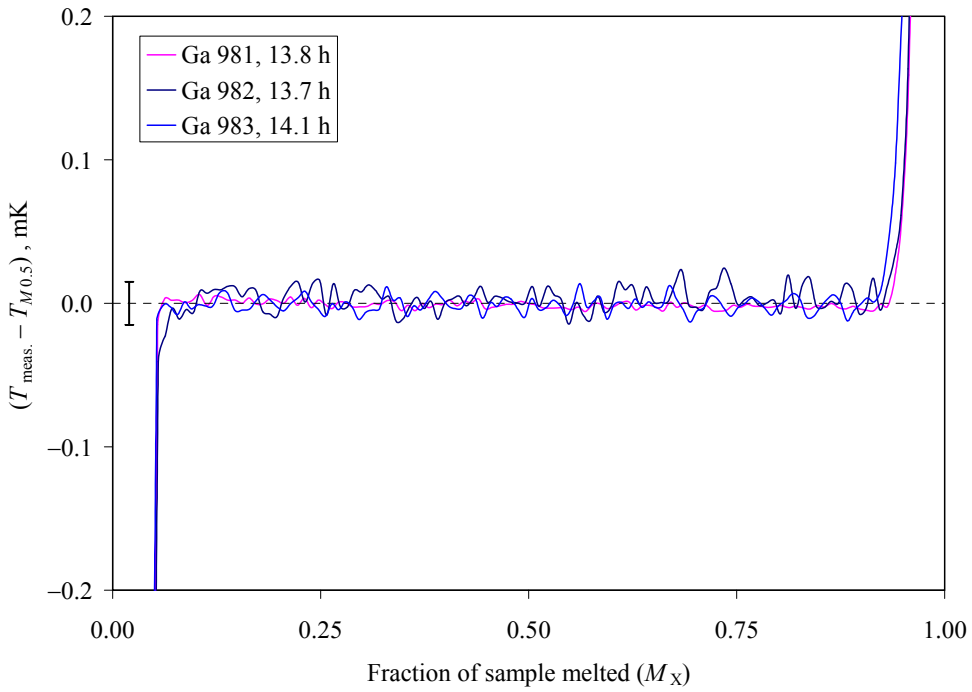

Figure 1. Representative melting curves for the Ga 981, Ga 982, and Ga 983 melting-point cells. The uncertainty bar (k = 2) represents the repeatability of the measurement system during the Ga MP realizations ($M_{0.1}$ to $M_{0.9}$). The duration of the melts is indicated in the legend.

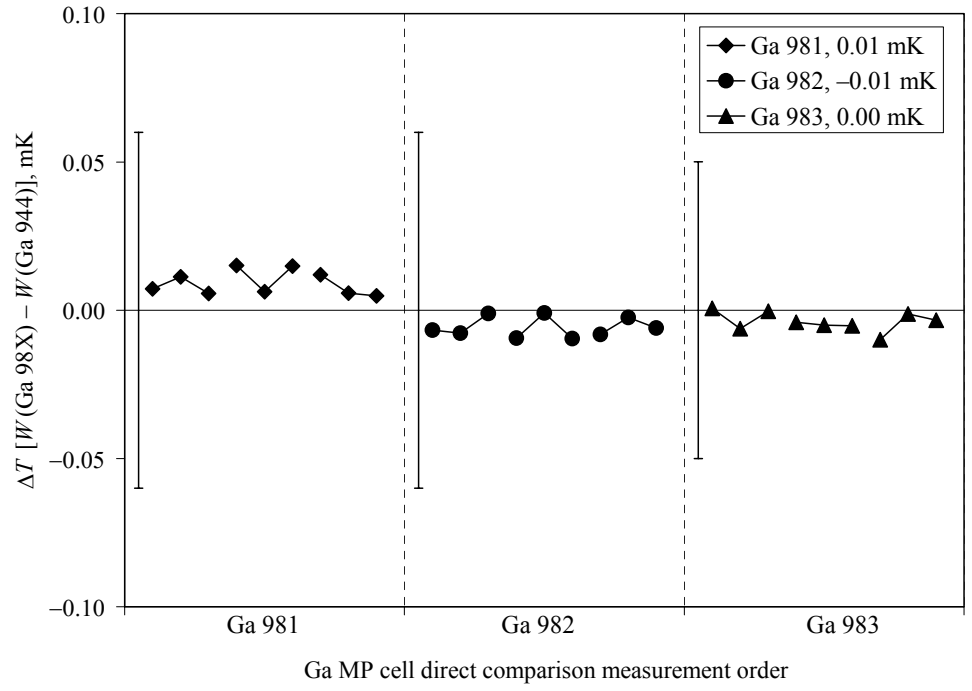

Figure 2. Direct comparison results of the realized melting-point temperatures for the Ga 981, Ga 982, and Ga 983 melting-point cells with respect to the NIST Ga MP reference cell Ga 944. The uncertainty bars (k = 2) represent the direct comparison measurement uncertainty for each cell paired with Ga 944. The average deviation of each cell from Ga 944 is indicated in the legend.

REFERENCES

[1] Preston-Thomas, H.; *The International Temperature Scale of 1990 (ITS-90)*; Metrologia, Vol. 27, pp. 3-10, p. 107 (1990).

[2] Strouse, G.F.; *Standard Reference Material 1751: Gallium Melting-Point Standard*; NIST Special Publication 260-157; U.S. Government Printing Office: Washington, DC (2004).

[3] Strouse, G.F.; *NIST Realization of the Gallium Triple Point*, The 7th International Symposium on Temperature and Thermal Measurements in Industry and Science – TEMPMEKO '99, NMi Van Swinden Laboratorium, Delft, The Netherlands; Dubbledam, J.F.; Groot, M.J., eds.; pp. 147-152 (1999).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.